REPORT OF PILOT-PLANT EXPERIMENTS ON THE RECOVERY OF VOLATILE FLAVOR FROM ORANGE JUICE

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Agricultural Research Administration
United States Department of Agriculture

December 1945

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SUMMARY

Pilot-plant studies were made on volatile flavor recovery from commercially produced orange juice by the process and the equipment which has been successfully applied to apple juice at the Eastern Regional Research Laboratory. Vaporization of 10 to 15 percent of the orange juice processed in a single-pass evaporator removed all of the organoleptically detectable volatile flavor. The volatile flavors of orange juice when condensed consist of an oil phase and a water phase. It was found that under atmospheric pressure operation the flavors in the water phase could be concentrated up to about a hundredfold without incurring any significant changes in its character or losses. As would be expected, the orange-oil recovered resembled distilled orange oil.

Thus far, the flavor recovery process has been operated at atmospheric pressure, and recovery of the fresh flavors without modification depends not only on a rapid vaporization but also on the stability of the volatile flavors at 212°F. when separated from the juice. Considering the heat sensitivity of orange juice, it is possible that the essences recovered would be of improved quality if the process were conducted under vacuum to lower the operating temperature. When conducted under vacuum, however, the loss of volatile flavors in the non-condensible gases discharged by the vacuum pump are much greater than the corresponding losses at atmospheric pressure. Thus, at 27 inches of mercury vacuum this loss is at least ten times the loss at atmospheric pressure. Means for recovering these losses are suggested in this report, and it is recommended that experimental studies investigating vacuum processing for volatile flavor recovery be made.

The orange essence, that is, the concentrated volatile flavors in the water phase, which was obtained in these studies is somewhat similar in ethereal character to apple essence; however, at the same relative concentration (referred to fresh juice) it is a much milder flavoring material. Its addition in the normal amount to a drink reconstituted from orange concentrate tends to mask the cooked flavor and other off-flavors which have developed on storage if these cooked flavors are weak. Tests now being conducted by the U. S. Citrus Products Station at Winter Haven will determine if this essence has sufficient value as an orange flavor to recommend its production and utilization.

When the stripped juice resulting from the flavor-recovery process was plated on dextrose-tryptone agar (pH 7.0) yeast colonies were not found, in most instances, but in the majority of samples colonies of bacteria were noted. When the process was carried out by effecting 12-1/2% vaporization in 9 seconds and cooling to 100°F. in 2 seconds by flash-vaporization into a chamber maintained at 2 inches of mercury absolute pressure no cooked flavor was observed in the stripped juice. This is significant in that it indicates that a concentrate base can be prepared suitable for the production of a full-flavored concentrate.

Suggestions for the design of 10-gallon-per-hour pilot plants, one for atmospheric pressure operation and one for vacuum are given in this report. The sizes of the evaporator and preheater recommended have been based on the heat transfer data secured in these experiments. These data have been tabulated in detail in the appendix for future reference.

This problem of recovery and utilization of volatile orange juice flavor is much broader than simply the perfection of the flavor recovery process. Obviously it should not be expected that a good flavoring essence as well as a high-grade juice concentrate suitable for use as a base for full-flavor concentrates can be obtained from low-grade juice. The juice which was used in these flavor recovery studies was obtained from nearby canners and since end-of-season fruit was being used, the juice was definitely not of high quality from a flavor standpoint.

A preliminary run was made on grapefruit juice. The data for this runare presented in an appendix of this report.

INTRODUCTION

The Eastern Regional Research Laboratory has developed a method for recovering and concentrating the fresh volatile flavors of apple juice as described in Circular AIC-63 and its Supplement Sheet.* From experiments on pasteurized Concord grape juice it was concluded that the volatile flavor recovery process could be applied successfully to grape juice. It was thought that the principles of the process should be applicable to other fruit juices and in the spring of 1945 plans were made for conducting flavor recovery experiments on orange juice at the Eastern Regional Research Laboratory using the pilot plant already set up.

This pilot plant has an operating capacity of 35 to 48-1/2 gallons per hour. Securing the quantity of fresh orange juice required for a run at the Eastern Regional Research Laboratory was found to be a difficult task. Local suppliers of fresh orange juice use hand reaming methods and did not have the necessary capacity. Juice obtained by pressing hand-peeled oranges in a rack and cloth press was judged to be distinctly inferior to that obtained by hand reaming. Furthermore, forty man-hours would have been required just to peel the oranges for producing 50 gallons of juice. When the experimenters at the Eastern Regional Research Laboratory realized how the character of the orange juice flavor depended upon the method of extraction they concluded that if the results were to be of any timely significance to the citrus industry they should be made on commercially produced orange juice; that is, juice produced by the canners on a large scale.

After consideration of the foregoing facts and of the interest that some of citrus people displayed by requesting that the Eastern Regional Research Laboratory's pilot plant be shipped to them at their expense for immediate studies, it was decided to ship the pilot plant to the U. S. Citrus Products

* The Supplement Sheet will be found at the end of the Appendix of the report.

Station at Winter Haven, Florida. This decision was made on April 23, and on May 15 the first orange juice run at Winter Haven was made. The experiments were conducted by H. P. Milleville of the Eastern Regional Research Laboratory, M. K. Veldhuis and G. N. Pulley of the U. S. Citrus Products Station, and E. M. Burdick of the Citrus Products Laboratory of Weslaco, Texas. The bacteriological analyses were made by Dr. Roger Patrick of the U. S. Citrus Products Station. Mr. C. F. Speh, Assistant Chief of Bureau of Agricultural and Industrial Chemistry was present at the first exploratory run, and Dr. L. F. Martin, Head of the Agricultural Chemical Research Division of the Bureau of Agricultural and Industrial Chemistry was present for the first three runs. The last pilot plant test on orange juice was made on May 28 when the season was practically over.

A test run on grapefruit juice with the pilot-plant unit was made several weeks later by M. K. Veldhuis and G. N. Pulley.

PRINCIPLES OF THE VOLATILE FLAVOR RECOVERY PROCESS

The process for volatile flavor recovery is a combination of four operations:

1. Vaporization of a fraction of the juice sufficient to remove all the volatile flavors;

2. Separation of the vapors from the unvaporized juice;

3. Fractionation distillation of the vapors to concentrate the volatile flavors;

4. Venting the non-condensible gases under conditions of temperature and pressure which reduce the loss of volatile flavors to a negligible quantity.

Thus far with apple and grape juices the operations have been conducted at or above atmospheric pressure. Recovery of the natural volatile flavors of juices without modification under these conditions depends not only on a rapid vaporization but also on the stability of the volatile flavors at 212°F. (the operating temperature of the fractionating column) when separated from the juice. In addition, if changes in the non-volatile flavors of the unvaporized stripped juice are to be avoided, the unvaporized juice must be cooled rapidly after separation from the vapors.

Loss of volatile flavoring constituents with the vent gas (the amount of non-condensible gases evolved from apple and grape juices amounted to 1% to 3% by volume of the juice processed) has been kept at a negligible quantity by:

(1) maintaining the partial vapor pressure of the volatile flavoring constituents in the vent gas to that corresponding to 70°F. or lower;

(2) limiting the concentration of the essence or concentrated volatile flavor distillate to 150-fold or less;

(3) venting at atmospheric pressure (as compared to vacuum operation).

Under these conditions with apple juice there has been a detectable aldehyde-like odor in the vent gas; the quantity of volatile flavors thus lost, however, is negligible. Thus, when the essence is blended with the unvaporized juice from which it was processed together with a quantity of water corresponding to that removed in the concentration of the volatile flavors, the blend so prepared is indistinguishable by organoleptic tests from the original unprocessed apple juice.

DESCRIPTION OF THE PILOT PLANT

The pilot plant has a capacity of up to 48-1/2 GPH of juice and is designed for atmospheric pressure operation. It is depicted in the accompanying diagram, Fig. 1. It was assembled from apparatus available at the Eastern Regional Research Laboratory and should not be taken as representative of the optimum design. Vaporization of the juice can be effected in three ways:

(1) single-pass evaporation without preheating;
(2) single-pass evaporation with preheating;
(3) superheating and flashing.

A description of the more essential items making up the pilot plant unit is given in the remainder of this section.

Metering Pump for Juice Feed: Two Milton Roy, stainless-steel, step valve, reciprocating pumps. The pumps were mounted in parallel, one having a capacity of 31 GPH, the other of 17-1/2 GPH. The pumping rate could be adjusted by means of a micro-indicating, worm-screw mechanism.

Preheater or Superheater: 18-1/2 feet (heated length) of 0.25 inch O.D., 0.18 inch I.D., (20 gauge wall) stainless-steel tubing.

Single-Pass Evaporator: 16-1/2 feet (heated length) single return bend of 0.75 inch O.D., 0.62 inch I.D. (16 gauge wall) stainless steel tubing. The evaporator was installed in a horizontal position between the preheater and the vapor-liquid separator as shown in Fig. 1.

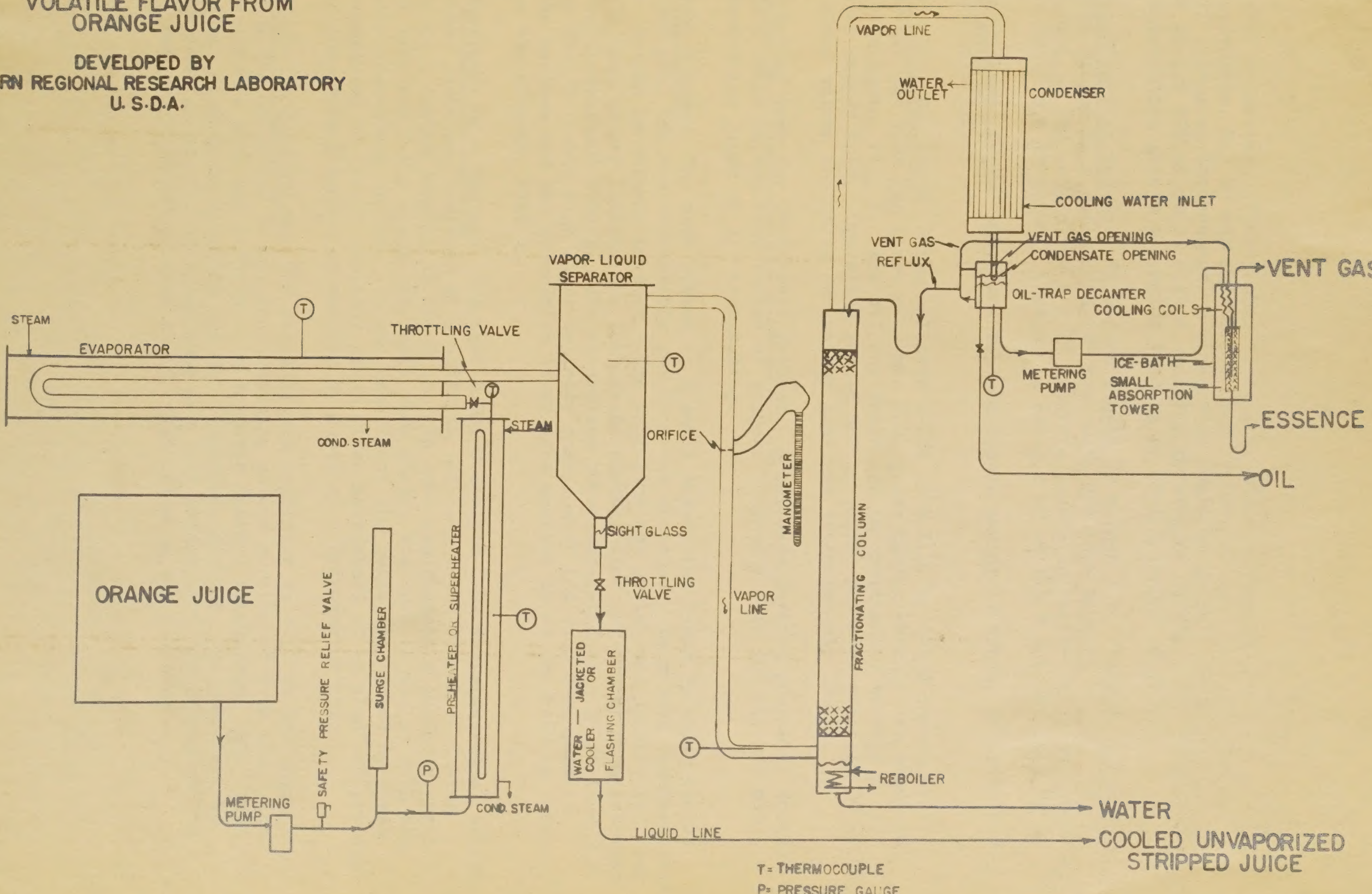
Vapor-Liquid Separator: Stainless steel cylinder 1 ft. dia. by 1-1/2 feet high. Vapor-liquid mixture enters at the midpoint as shown in Fig. 1 and is deflected downwards by a short baffle placed at a 45° angle to the horizontal.

Vapor-Line Orifice and Manometer: In the vertical section of stainless steel pipe (1-inch I.P.S.) which conducted the vapors from the vapor-liquid separator to the fractionating column a 0.40-inch diameter, thin-plate orifice was inserted at a union fitting. From pressure taps located on both sides of the orifice, simple vapor-line connections were made to a

Fig. 1

DIAGRAM OF PILOT PLANT
FOR RECOVERING
VOLATILE FLAVOR FROM
ORANGE JUICE

DEVELOPED BY
EASTERN REGIONAL RESEARCH LABORATORY
U. S.D.A.



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water manometer. These connecting lines would occasionally fill up with condensate, but by manipulation of vent valves on the manometer and shut-off valves on the connecting lines they could be quickly refilled with air. At 48-1/2 GPH of juice with a specific gravity of 1.045, a 10% by weight vaporization gave a manometer reading of 30-1/2 inches of water.

Fractionating Column: Five-foot length of 4-inch I.D. flanged, pyrex-glass pipe packed to a depth of 3-1/2 feet with 3/8-inch Raschig rings. At the bottom of the column was a 2 KW electric immersion heater which was operated at about 0.6 KW input. (Heater is rated 2 KW at 115 V but was used at 50 V).

Condenser: Copper shell and tube condenser having 17 tubes, 5/8-inch O.D., 1/2-inch I.D.

Oil Trap Decanter: Glass cylinder 1-1/4 inch I.D. by 6-1/2 inches high. Reflux and vent gas lines of 9 mm. I.D. glass tubing arranged as shown in the diagram, Fig. 1. The oil was drawn off intermittently through a 1/8-inch copper tube. Operating liquid holdup volume was approximately 90 ml.; holdup volume including the reflux line was approximately 130 ml.

Metering Pump for Condensate Product: A bellows-operated pump with double-ball check valves constructed of copper. Capacity 5 to 100 ml. per min. Connecting lines of 1/8-inch tubing.

Vent System: The vent system is a small absorption tower in which cooled vent gas, the non-condensible gas from the condenser, is passed countercurrent to cooled condensate product. The vent line and cooler was constructed from 1/4-inch copper tubing, the condensate product line and cooler from 1/8-inch stainless-steel tubing. The absorption tower consisted of a 1-1/8-inch I.D. by 10-1/2 inches high glass cylinder packed with 1/4 inch Berl saddles. The coolers and the absorption tower were mounted in an ice bath as shown in the diagram, Fig. 1.

Stripped-Juice Cooler: 5/8-inch O.D., 0.53-inch I.D. stainless-steel tubing, water jacketed, composed of 3 units in series. Total length of tubing 22 ft.; cooled length, 18 ft.-0 in. In some experiments a 10-foot length of 1/2-inch I.D., block tin, coil worm cooler was used in series with the stainless-steel cooler. In one experiment the juice was cooled by flash vaporizing it into a 12-liter glass flask maintained at an absolute pressure of less than 2 inches of mercury by means of a Hydrovactor pump.

Adiabatic Insulation: The vapor-liquid separator as well as the vapor line from the separator to the fractionating column were wound with electrical resistance wire and covered with magnesia thermal insulation. The fractional distillation column was not insulated, however, the reboiler was operated so as to just compensate for heat losses from the fractionating column and vertical section of the vapor line to the condenser.

PROCEDURE

Preceding each run the pilot-plant unit except for the juice cooler was brought into thermal equilibrium by running on water for at least half an hour under the conditions proposed for the juice. (When ice-cooled water was used to cool the stripped juice it was circulated through the juice cooler only when samples were taken in order to conserve ice.) When the evaporator was used to effect vaporization of the juice the rate of vaporization was controlled by regulating the steam pressure to the evaporator steam chest so as to maintain a constant pressure drop across the vapor-line orifice. If the preheater was used with the evaporator the steam pressure in the preheater chest was regulated so as to maintain the desired juice temperature as measured by the thermocouple in the juice line leaving the preheater. When the preheater was used as a superheater the throttling valve (see Fig. 1) was regulated so as to maintain the juice in liquid phase in the preheater and the steam pressure was regulated so as to maintain a constant superheat temperature. The superheated juice was flashed into the vapor-liquid separator by way of the evaporator tubing which was maintained adiabatic by keeping about 2 pounds steam pressure in the evaporator chest. (Note at 48-1/2 GPH and 10% flash vaporization the time required for passage of the vapor-liquid mixture through the evaporator is only 1/6th of a second.)

The vapor-liquid separator and the vapor line from it to the fractionating column were operated practically adiabatically by regulating the electrical input to their jacket windings until thermocouples in the separator and vapor line gave readings slightly above the boiling point of the juice at the operating pressure--atmospheric pressure plus the fluid-flow pressure drop. The operating characteristics of the fractionating column were not studied; the reboiler was regulated so that the rate of reboiling equalled the heat losses from the fractionating column and the vertical section of the vapor line to the condenser (approximately 0.17 gallons of water were vaporized per hour by the reboiler).

The product metering pump was set at 1/Xth the fresh-juice feed rate where X represents the strength or fold of the essence desired as compared to fresh juice. At the beginning of the juice run the pump was shut off and the fractionating column was run on total reflux until the composition of the condensate and reflux was estimated to be that of the desired product, after which the pump was started again. The length of time that the column was run on total reflux was calculated by dividing the effective hold-up volume of the fractional distillation system (90 ml. for the decanter plus 40 ml. for the reflux return line plus 50 ml. for the estimated equivalent of reflux in the column at product strength, making a total of 180 ml.) by the condensate product pumping rate. Thus, for a fresh juice feed rate of 48-1/2 GPH, the time on initial total reflux operation is found to be 6 minutes when a 100-fold (by weight) essence is produced, (specific gravity of juice assumed to be 1.04).

When the objective of a run was simply to study the completeness of the removal of volatile flavors from the juice or of the modification of flavors induced by the processing, the fractionating column was in effect removed

from the system by not returning condensate to it. The reboiler was operated as already described to compensate for heat losses from the fractional distillation system. The vapors were thus conveyed without rectification to the condenser, where they were condensed and collected.

The orange juice for all of the runs was obtained from nearby canneries all of whom use the Faulds rotary-press juice extractor. The quantities so obtained were approximately as follows:

Bordo	10 gals.
Pasco	65 gals.
Polk Packing Assn.	350 gals.
Total	425 gals.

The juice obtained for this work was from end-of-season fruit and poor from a flavor standpoint. The acid and soluble solids contents were low and the fruit used was well past optimum maturity. The juice was inferior in flavor in comparison with hand reamed juice from oranges obtained earlier in the season and kept in cold storage. There is room for improvement in the quality of commercially expressed juice and studies at Winter Haven, Florida and Weslaco, Texas have as an objective the improvement in flavor of the freshly expressed and processed juices.

Except for samples and some 20 gallons which were used to produce concentrate, the processed or stripped juice was discarded. For this reason unless large quantities of processed juice (or essence) are desired, as was the case with apple juice, it is recommended that future experimental studies be conducted on a smaller capacity pilot plant. A 10 GPH unit is probably about the smallest size that can be operated without special "micro" technics. Thus at 10 GPH the rate at which 200-fold essence is withdrawn is only 3.3 ml. per minute.

RESULTS AND DISCUSSION

Flavor Changes During Processing:

Previous experience on apple juice with this pilot plant unit has indicated that of the three methods for vaporizing juice--evaporator, evaporator with preheater, or superheater--the evaporator method has the least heating effect on the juice (an evaporator with a preheater would be just as good if the length of the evaporator tubes were shortened). Each of the three methods was tested on orange juice, but the results were the same as for apple juice. The stripped juice resulting from processing fresh juice had a slight cooked flavor and aroma when cooled by the stripped juice cooler and block tin coil, but not when cooled nearly instantaneously by flashing into a vacuum. Data corresponding to these conditions are given in Table I. It is recommended that in future studies at atmospheric pressure an evaporator be used which will effect the vaporization in a shorter time--at least half the present time--and that the stripped juice be cooled rapidly, either by flashing into a vacuum or by a more efficient cooler. A suggested design for a 10 GPH pilot plant is given in Appendix I.

TABLE I

Vaporization	Cooling of Stripped Juice	Flavor of Stripped Juice
10 to 12.5% in 15 seconds in evaporator 10% in 8 seconds in evaporator after being preheated to 217°F. in 2 seconds. 8% by superheating to 282°F. in 2½ seconds and flashing to atmospheric pressure.	By water jacketed cooler followed by a worm cooler using ice water as cooling medium taking 35 seconds to cool juice to 90° F.	Slight cooked odor and Slight cooked taste
10 to 12.5% in 15 seconds in evaporator	By flash-vaporization into a chamber maintained at 2 inches mercury abs. pres. (100°F.) within 2 seconds after leaving separator	Odorless No cooked taste

When the vapors removed from orange juice were condensed a two phase condensate was obtained. The flavor of the oil phase was judged to be very similar to what is known in the trade as distilled orange oil. Whether or not the flavors found in the water phase of the condensate have been changed is not definitely known. It would appear that any changes are of a minor nature and do not resemble the changes that develop in canned orange juice or concentrate in storage. If 100-fold orange essence (water phase) were diluted with (odorless) distilled water back to juice strength a product was obtained having an odor very similar to that of oil-free juice. (The oil-free juice was obtained by hand-peeling an orange and then squeezing the juice out of the peeled orange). The slight differences noted might have been the result of the heating or they might have been due to volatile, water-soluble, flavoring constituents present in the commercially expressed juice which were not in the laboratory-expressed juice. No final conclusion is possible at this time.

The volatile oil collecting in the oil trap decanter was judged to resemble what is known to the trade as distilled orange oil. Citrus peel oils are composed of mixtures of a number of different compounds of varying volatility. Generally 90% and more is limonene. The limonene contributes nothing to the flavor and it is more volatile than most of the portions containing the characteristic flavor and aroma. It is common practice in the industry to remove most of the limonene by fractional distillation under vacuum and market the residue as vacuum concentrated oil. The

oil recovered was similar to that recovered from vacuum citrus juice "de-oilers." There is some possibility of alteration in flavor of the volatile oils during the essence recovery process, but it would appear that any changes were of a minor nature.

It is recommended that at least some of the future studies on the volatile flavor recovery of orange juice be made processing under vacuum so that none of the flavors present is modified. The products from such a process would furnish an excellent criterion for judging products obtained under other conditions, and may be valuable enough to make the vacuum process feasible for commercial adoption. Vacuum processing for volatile flavor recovery, however, will not be successful unless special means are taken to avoid excess loss of volatile flavors which escape with the non-condensible gases vented out the vacuum pump. The factors concerned with vent losses are discussed in detail in Appendix III. A suggested design for a 10 GPH pilot plant for vacuum operation is given in Appendix II.

Removal of Volatile Flavor

Since the peel oil is the dominant flavor present and to a considerable extent masks the other volatile flavors, and since a modified or cooked odor was induced in the unevaporated stripped juice unless it was cooled rapidly, it is difficult to conclude with accuracy what percent vaporization was required to remove all of the organoleptically detectable volatile flavors. Thus, in one experiment the percent vaporization was varied from 5% to 7-1/2% to 10%. The stripped juice from 5% vaporization had more odor than the stripped juice from 7-1/2% vaporization, which in turn had more than the stripped juice from 10% vaporization. In the 10% stripped juice only a very slight cooked odor was noticeable, whereas in the other stripped juices a fresh juice character was faintly noticeable. From these observations it was concluded that 10% vaporization is approximately the minimum required for removing organoleptically detectable, fresh orange, volatile flavor and that until more accurate tests can be made 10 to 15 percent would be recommended as the vaporization necessary to remove all of the volatile flavors.

Peel-oil analyses were made of the fresh juice and stripped juice for most of the runs and are presented in Table II. How the percent oil removed varies with the percent vaporized is shown in Fig. 2.

TABLE II

	Peel-Oil Analyses - %			% Vaporized
Expt.No.	Fresh Juice	Stripped Juice	% Oil Removed	(Single-Pass)
2	0.024	0.007	58	7.6
3	0.029	0.009	69	8.5
4	0.019	0.007	63	10.1
	"	0.008	58	7.9
	"	0.010	47	5.3
5	0.041	0.011	73	12.5
6	0.029	0.007	76	12.5

Maximum Concentration and Nature of Essence Produced

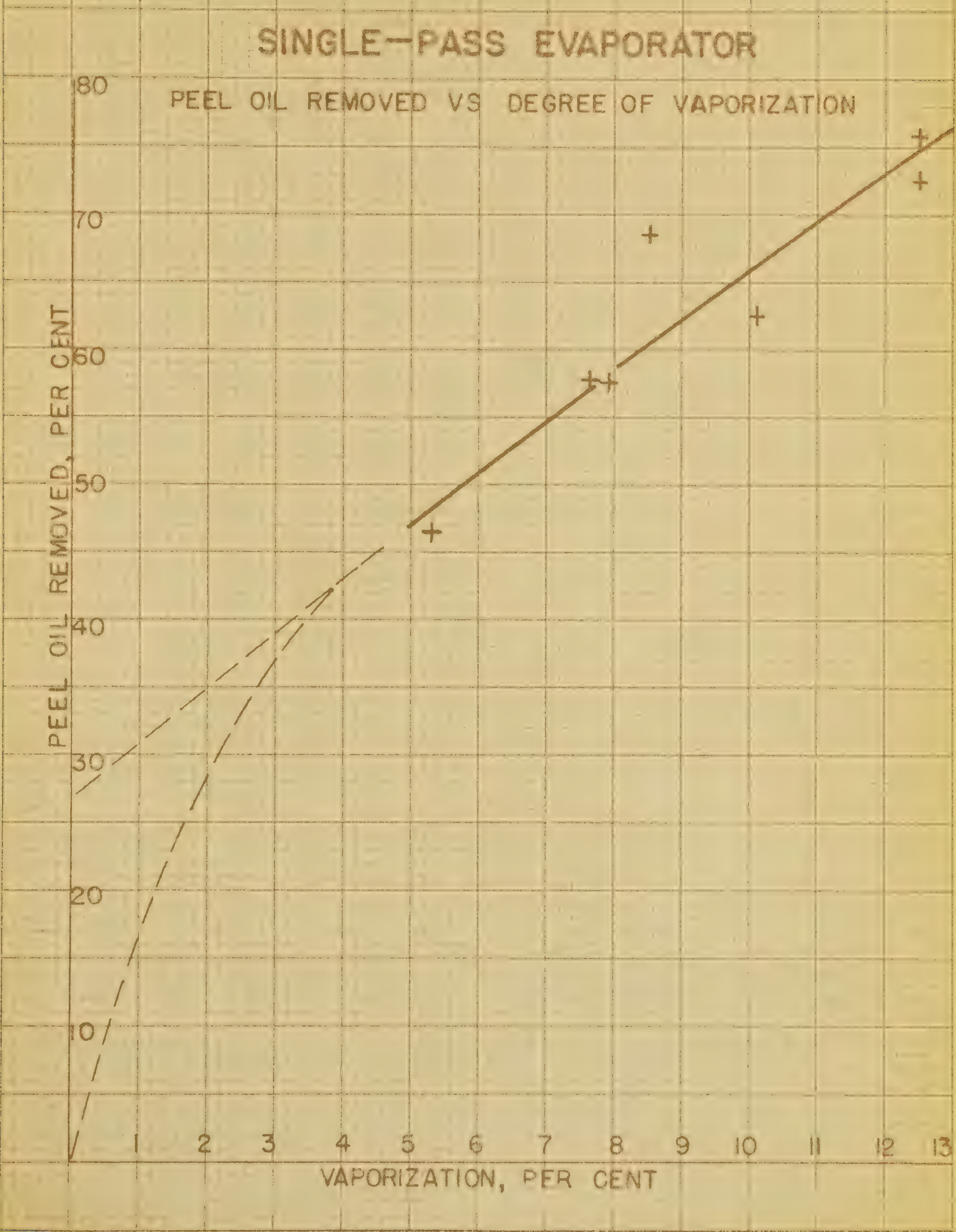
The term essence is used throughout this report to denote the water phase of the condensate product. In one experiment, essences with a theoretical concentration of one hundredfold, two hundredfold and four hundredfold were prepared. When these essences were diluted back to juice strength with (odorless) distilled water and compared it was discovered that the sample prepared from the hundredfold essence was noticeably stronger than the others. Since the hundredfold sample was in odor the same strength as the oil-free orange juice described previously it was concluded that it was possible to produce a hundredfold essence without loss but not a 200-fold essence. That is, when it was attempted to prepare a 200-fold essence losses occurred from the flavor recovery system. Furthermore, since all the bottoms products from the fractionating column for the three essences had negligible odor it was concluded that the losses occurred in the non-condensible vent gases.

The vent-gas rate for all of the orange juice processed was rather constant and approximately 5% by volume or about twice that observed for apple juices processed at the Eastern Regional Research Laboratory. In the experiment just described it was cooled and vented through ice-cold essence in the vent system, which was immersed in an ice bath. This vent gas had a rather pungent aldehyde-like odor closely resembling the vent gas odor obtained when processing apple juice, but in Milleville's estimation was stronger. (Orange essence itself is quite similar in ethereal character to apple essence, but at the same relative concentration referred to the fresh juice it is a much milder flavoring material.)

The venting rate with the particular orange juice processed was about twice that previously experienced on apple and grape juices. Since the vent loss is proportional to the quantity of non-condensible gas vented (cf. Appendix III), and the volatile flavors in the vent gas obtained are quite similar, it should be expected that the maximum concentration of essence obtainable would be less for a juice which evolves a larger quantity of non-condensible gas. With apple juice the maximum concentration of essence obtainable without significant loss is between 150 and 200-fold. Most of the apple juice processed had a vent gas rate of 2 to 3 percent by volume of the juice feed. It is to be expected, therefore, that for a juice, such as the orange juice which we processed, which evolves 5 percent by volume of vent gas, the maximum concentration of essence producible without loss should be about a 100-fold. It should be noted that since the essence is "distilled water" plus an estimated few tenths of one percent flavor constituents it is not practical to reduce the vent losses by using a lower venting temperature than the freezing point of water.

The interest of our studies was primarily in the volatile, non-oil flavor, yet the dominant flavor in the fresh juice which we processed was contributed by the oil and masks the other volatile flavors present. The flavor of the orange essence obtained in these studies is weak compared to orange oil. Furthermore, although the essence is practically oil-free it is nevertheless saturated with the oil, having been in intimate contact with it. As one result, a sample prepared by diluting 100-fold essence to juice strength will have twice as much oil as a sample prepared by diluting a

FIG. 2



SINGLE—PASS EVAPORATOR
PEEL OIL REMOVED VS DEGREE OF VAPORIZATION
PEEL OIL REMOVED, PER CENT
80
70
60
50
40
30
20
10
1
2
3
4
5
6
7
8
9
10
11
12
13
VAPORIZATION, PER CENT

200-fold essence to juice strength. Because of these complications it is difficult to conduct reliable organoleptic tests with orange essence. Hence, some of the conclusions presented in this report are not as definite as one would like them to be.

Utilization of the Orange Essence

The final report on the value of the orange essence produced in these experiments must await the results of tests being conducted at the U. S. Citrus Products Station in Winter Haven. (See Appendix VII for the first report on these tests). Even when these results become available, the possibility that future studies will produce an essence with superior characteristics should be kept in mind. The only application which was immediately tested with the essence which was produced was in the preparation of a full-flavor concentrate. Addition of the essence to orange juice concentrates tended to mask the canned or cooked flavors present if these modified flavors were not strong. The results, however, were not outstanding and not detectable in the average grade of commercial concentrate in which these modified flavors are rather strong. The objective of improving commercial orange concentrate by the addition of recovered essence so that it is a satisfactory, full-flavored concentrate constitutes an important subsidiary problem to the volatile flavor recovery studies.

Microbiological Analyses of Fresh and Processed Juice

Microbiological analyses of fresh juice and stripped juice were made on five runs. The average counts were as follows:

Tryptone dextrose acid agar (pH 3.3)	95,000
Tryptone dextrose agar (pH 7.0)	225,000
Wort agar (pH 5.0)	285,000

The majority of the organisms in the fresh juice were yeasts, but considerable numbers of bacteria were also present. These counts are not surprising considering the condition of the fruit and that the prevailing temperature was favorable to the growth of microorganisms in the juice and on cannery equipment.

In every case when processed juice was plated on tryptone dextrose agar some colonies were noted. With wort agar and tryptone dextrose acid agar a few colonies were noted on some plates. Even though the number of organisms on the plates was not sufficient to give accurate counts, the juice could not be considered sterile. The majority of the organisms in the processed juice were bacteria and their presence may or may not be of significance. The results on the stripped juice were not entirely satisfactory because it was necessary that the samples be taken from the end of the discharge hose. This hose had to be handled in changing the stripped juice receivers and the few organisms found might have come from the outside of the hose rather than from the stripped juice.

It seems reasonable to assume that the cooked flavor development is a progressive one, and that the time taken before the cooked flavor can be detected depends on the temperature level. One would also expect that the

effect of successive heat treatments should be additive. Thus, two heat treatments, one of which might be simply storage at room temperature, neither of which would develop a cooked flavor in the juice might develop the cooked flavor if applied successively. It would therefore seem desirable to conduct further studies to determine the minimum heating conditions which result in sterilization. Other operating conditions remaining the same, such a study would involve the use of an evaporator which would effect the vaporization in a shorter time. The design suggested for such an evaporator having a juice feed capacity of 10 GPH is considered in Appendix I.

Heat Transfer Performance Data

It is regrettable that lack of time has thus far prevented the inventor of the volatile flavor recovery process from making an analysis of the extensive pilot-plant data he has accumulated at the Eastern Regional Research Laboratory on the processing of apple juice for volatile flavor recovery. Such an analysis would have formed a basis for judging the reliability of some of the correlations presented in this report. It was observed, however, that orange juice was slightly easier to heat and vaporize than apple juice. Thus, for the same flow conditions the heating steam temperatures of the preheater as well as the evaporator were a few degrees Fahrenheit lower for orange juice than for apple juice. For future reference the thermal data taken during each orange juice experimental run is tabulated in Appendix V.

The first fact to take note of in considering the analysis of the data presented is that although the log mean temperature difference was used for computing heat transfer coefficients, its use under the conditions encountered involves an error. Theoretically, log mean temperature differences should be used only when the coefficient of heat transfer does not vary with temperature. Consider the preheater data in Table IV for two orange juice runs in which the only variable was the final temperature to which the juice was heated, and note the variation in the overall heat transfer coefficient.

TABLE IV

	Temperatures			Overall Heat
Juice Rate	Juice In	Juice Out	Steam	Transfer Coefficient
39.5 GPH	91°F.	282°F.*	297	1040
39.5 GPH	91°F.	210.5°F.	223	750

*The pressure on the superheated juice was 30 p.s.i. in excess of that required to maintain it in the liquid phase.

No doubt the reason for this variation is that the viscosity of the juice decreases considerably with the increase in temperature, and as a result the juice is easier to heat.

In order to correctly analyze such data the value of the heat transfer coefficient must be known as a function of the temperature. With the data at hand this is neither known nor calculatable. This error in the use of the log mean temperature difference becomes important when coefficients so computed are used for a full temperature range differing from those under which they were computed. Since in most of these experiments the full temperature range was the same, the correlations presented should be fairly reliable but only for the full temperature range indicated.

The variation of the preheater overall coefficient of heat transfer with juice velocity is given in Fig. 3. According to theory, the juice film coefficient varies as the 0.8 power of the velocity. To the juice-film, heat transfer resistance, however, must be added the resistances of the metal wall and steam film. Since these other resistances are constant it is to be expected that the overall coefficient U will vary to a lesser power of the velocity than 0.8, which is the case as shown in Fig. 3.

Nomenclature

T mean temperature difference, $^{\circ}F$.
A effective heat transfer area based on the I.D. of preheater and evaporator tubes, sq. ft.
Q heat transfer rate, (BTU)/Hr)
U overall heat transfer coefficient, $\frac{(BTU)}{(Hr.)(Sq.Ft.)(^{\circ}F.)}$
h juice film heat transfer coefficient, $\frac{(BTU)}{(Hr.)(Sq.Ft.)(^{\circ}F.)}$
V velocity, feet/sec.
D diameter, feet
Basic heat transfer equation; Q = UAT

Also according to theory, h, the juice film coefficient of heat transfer, varies as $1/D^{0.2}$ or combining these variables h varies as $\frac{V^{0.8}}{D^{0.2}}$. It is interesting therefore, to compare the U obtained for the 0.18 inch I.D. juice preheater with the U for the 0.53 inch I.D. juice cooler. Theoretically it is found that the juice film coefficient for the cooler should be 85% less than the juice film coefficient for the preheater. Data for computing U for the cooler is incomplete, but it is estimated (see Appendix VI) that at a juice velocity of 1.1 ft./sec. U for the cooler is about 250. For the preheater at a juice velocity of 10 ft./sec. U is 830. U for the cooler is thus 70% less than U for the preheater which is in good agreement with theory.

All of our runs except two short ones were made using the evaporator without the preheater. In other words, the preheating of the juice to its boiling point as well as the vaporization is accomplished in the evaporator tube. In this connection it should be noted that below 13% vaporization more than half of the heat transferred in the evaporator tube is preheat--see Fig. 4.

The variation of the evaporator heating steam temperature with percent vaporization for a constant juice feed-rate is shown in Fig. 5. In Fig. 6, the percent vaporization is constant at 10% and the variation of steam temperature with juice throughput is shown. Also shown on Fig. 6 is the lowering of the steam temperature by 22°F. when juice at the rate of 48.5 GPH was preheated to 217°F. (its boiling point at the pressure existing in the evaporator).

Operation of the Evaporator without a Preheater

Before further considering the evaporator data just presented, attention is called to the unusual application it represents. In most conventional evaporators the maximum evaporation achieved per pass is only 2 to 3% or only a fifth of what is obtained in this pilot plant evaporator. Moreover, it is customary to use a preheater with continuous evaporators, whereas with this pilot-plant evaporator best results as measured by the severity of the heat treatment given a heat sensitive juice are achieved without the use of a preheater.

Tests made measuring the time taken for colored water to pass through the evaporator operated without a preheater indicate that when vaporizing 10% at 48.5 GPH, approximately one-third of the volume of the tube is occupied with vapor. From this observation it is concluded that the first two-thirds of the evaporator tube preheats the juice to its boiling point and that only the last third of the tube functions as an evaporator.

On the basis of severity of heat treatment, juice-flow pressure drop, fouling, heating steam temperature required, and capacity, the optimum operating conditions for this evaporator without a preheater are obtained in the range of 10% to 15% vaporization when processing 48.5 GPH of juice. Since only a third of this evaporator tube actually functions as an evaporator it is concluded that when a preheater is used with this evaporator the tube length ought to be reduced to a third of its present length to give comparable optimum results. In fact, it is quite possible that shortening the present evaporator tube from 16-1/2 feet to less than one-third its length and operating it with a preheater would give superior results. For a given installation the use of a preheater means an extra item of equipment and more temperature controls; however, these disadvantages may be outweighed, particularly for large-scale operation, by the shorter length of evaporator tube required.

Coefficients of heat transfer for the evaporator operating without a preheater have not been discussed thus far because it is not possible to compute them according to standard methods from the data which was taken. Thus, T in the equation Q = UAT varies from 180°F. to 40°F. in the preheater section of the evaporator and then remains constant at 40°F. A method has been developed, however, for computing an evaporator coefficient which is thought to be a useful analytical tool and deserving of study. It is presented in Appendix IV.

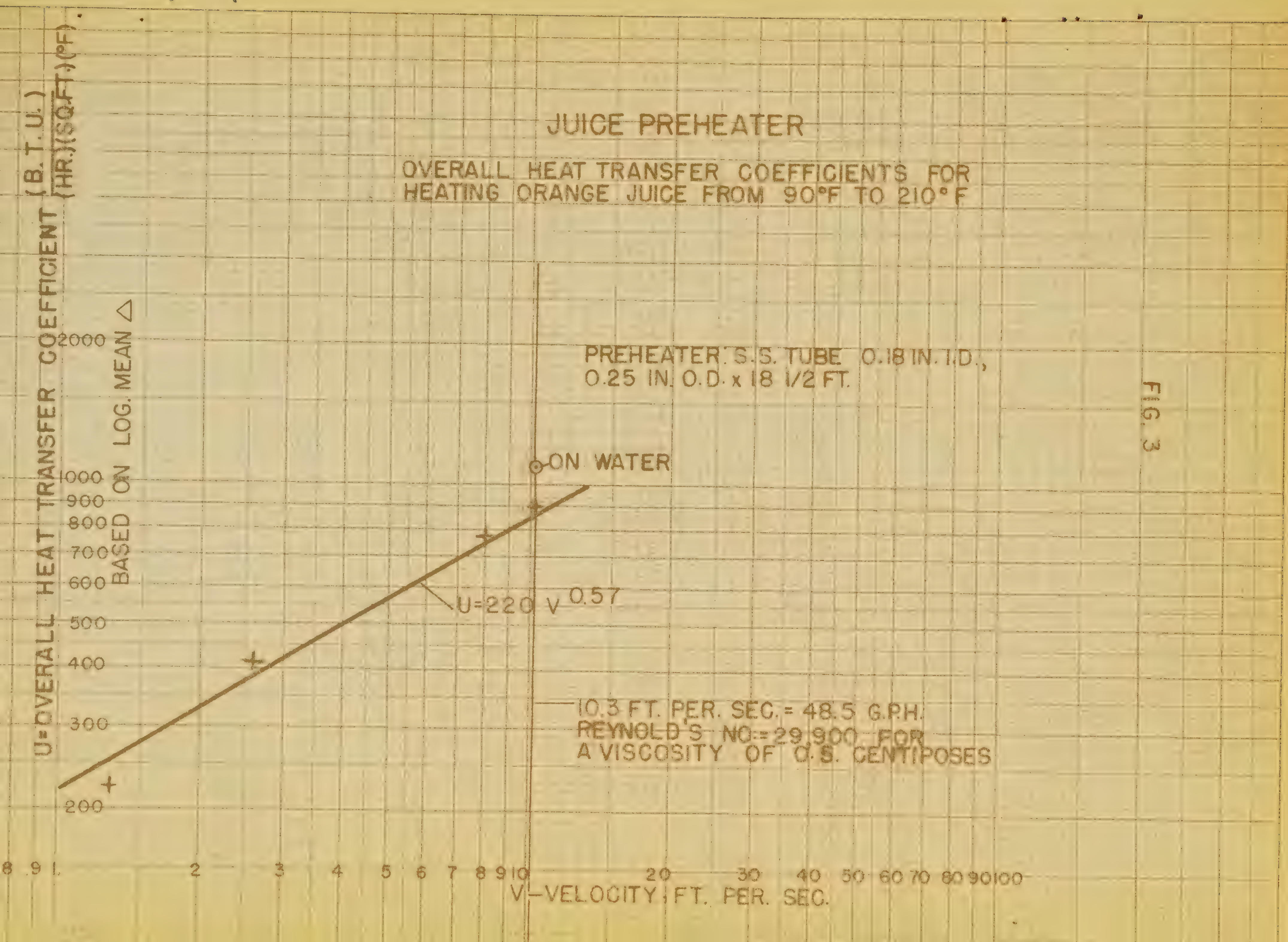

JUICE PREHEATER
OVERALL HEAT TRANSFER COEFFICIENTS FOR HEATING ORANGE JUICE FROM 90°F TO 210°F
PREHEATER: S.S. TUBE 0.18 IN. I.D., 0.25 IN. O.D. x 18 1/2 FT.
ON WATER
U=220 V 0.57
10.3 FT. PER. SEC. = 48.5 G.P.H.
REYNOLD'S NO.=29,900 FOR A VISCOSITY OF 0.8. CENTIPOSES
U=OVERALL HEAT TRANSFER COEFFICIENT (B.T.U.)/(HR.)(SQ.FT.)(°F)
BASED ON LOG. MEAN △
2000
1000
900
800
700
600
500
400
300
200
8 9 1 2 3 4 5 6 7 8 9 10 20 30 40 50 60 70 80 90 100
V–VELOCITY, FT. PER. SEC.

FIG. 3

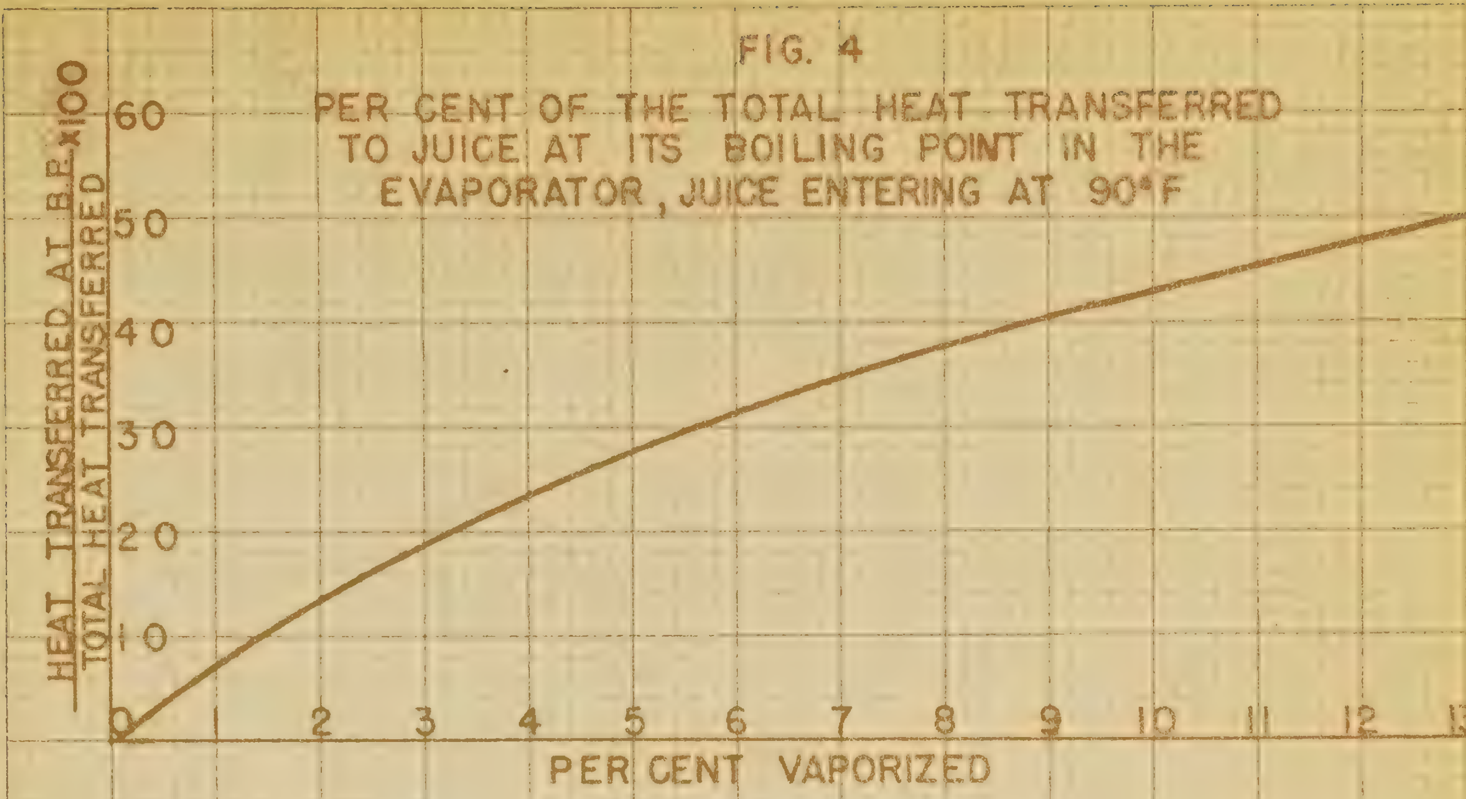


FIG. 4
PER CENT OF THE TOTAL HEAT TRANSFERRED TO JUICE AT ITS BOILING POINT IN THE EVAPORATOR, JUICE ENTERING AT 90°F

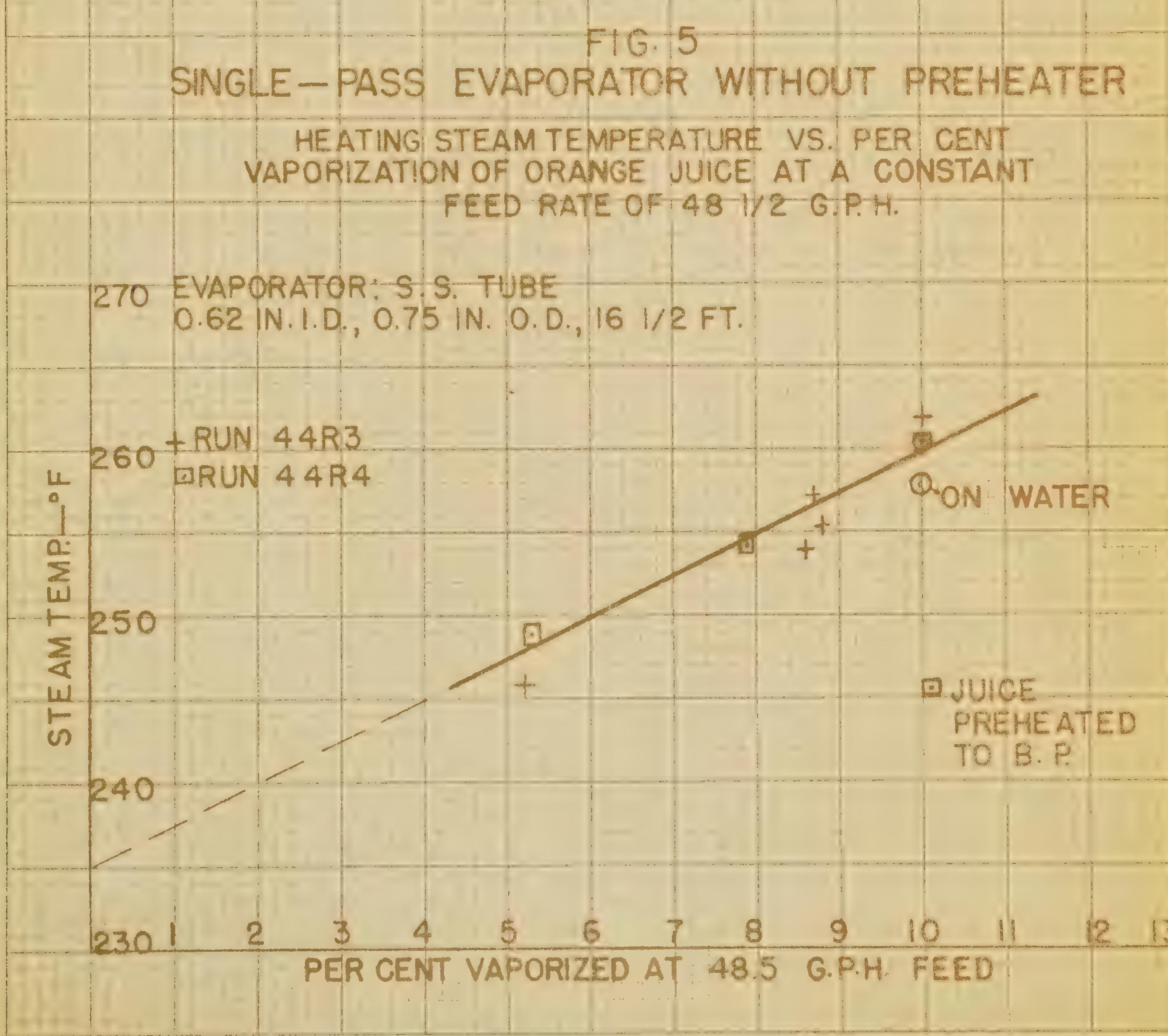
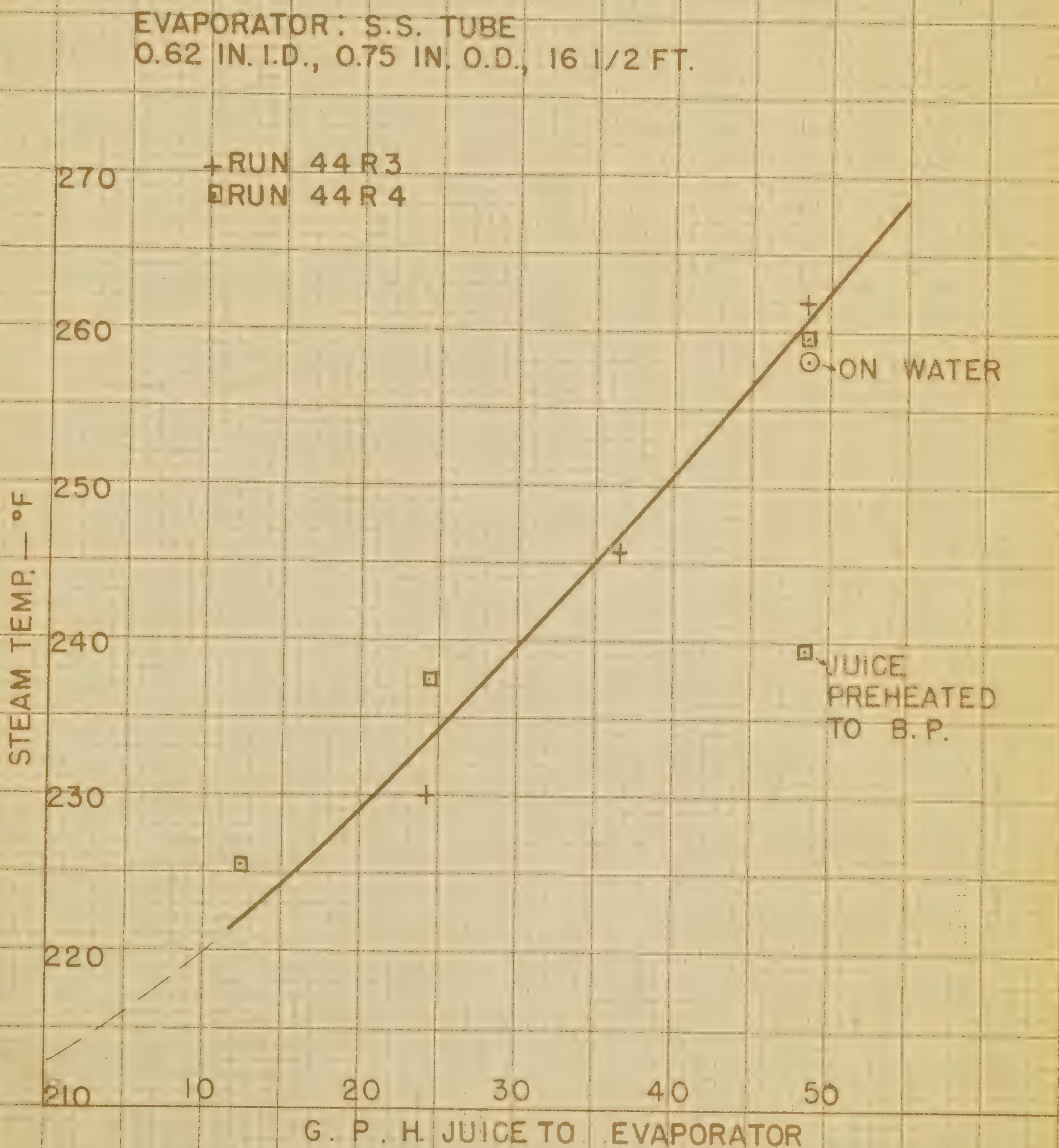


FIG. 5
SINGLE-PASS EVAPORATOR WITHOUT PREHEATER
HEATING STEAM TEMPERATURE VS. PER CENT VAPORIZATION OF ORANGE JUICE AT A CONSTANT FEED RATE OF 48 1/2 G.P.H.

FIG. 6

SINGLE-PASS EVAPORATOR WITHOUT PREHEATER

HEATING STEAM TEMPERATURE VS. ORANGE JUICE FEED RATE AT A CONSTANT PER CENT VAPORIZATION OF 10%



CONCLUSIONS AND RECOMMENDATIONS

1. Orange essence up to a strength of about 100-fold can be produced without a significant loss when processing at atmospheric pressure by the volatile flavor recovery process developed for apple juice.

2. It is possible to remove all of the volatile flavor from orange juice without developing a cooked flavor in the processed juice; however, the time of heating used was so critical that the juice had to be cooled nearly instantaneously by flashing to a vacuum. It is recommended that if further experiments at atmospheric pressure are undertaken the heating time be shortened as much as possible.

3. It is not known whether or not the orange essence secured by the process operated at atmospheric pressure was modified by the heating or not. If there was any modification of the essence non-oil flavors, it was judged to be of a minor nature although not necessarily an unimportant one.

4. When the volatile flavor recovery process is operated at atmospheric pressure on orange juice, noticeable losses of volatile flavor in the vent gases begin to occur at an essence concentration of about a hundred fold. These vent losses would be increased markedly if vacuum operation of the process were attempted in order to make certain that none of the flavors are heat-modified in the process. For vacuum operation to be successful steps must be taken to recover these losses, or the process must be modified to avoid them. Suggestions for overcoming the losses of volatile flavor when operating under vacuum are given in this report.

5. The use of the orange essence secured in these experiments for improving the flavor of orange concentrate was not particularly successful because of the mildness of the orange essence flavor and the strength of the modified orange flavors present in the juice concentrate. It is recommended that studies by others on the improvement of the flavor of orange juice concentrate, particularly that produced commercially, be continued.

6. Tests now in progress at the U. S. Citrus Products Station, Winter Haven, will give additional and more conclusive data on the utilization of orange essence. Whether or not the results of these tests indicate that the orange essence secured in these experiments is of utility, it is believed that further studies are warranted along the lines indicated.

Appendix I

10 GPH PILOT PLANT FOR OPERATING UNDER ATMOSPHERIC PRESSURE

Design Suggestions

Resume

Evaporator without Preheater--See Fig. 7 for dimensions. Evaporator with Preheater--Use 1/3 length given in Fig. 7 for evaporator, and Fig. 9 for preheater dimensions.

Fractionating column--2 in. I.D. by a 3-1/2 ft. height of 1/4 inch Raschig ring packing.

Operating Conditions

The designs given are based on processing 10 GPH of juice and vaporizing 12.5% by weight. The heating steam temperatures required should approximate those used in the 48.5 GPH unit; however, they may have to be adjusted somewhat to compensate for any errors in the assumptions upon which the design calculations are based. When an evaporator is used with a preheater, it is important to note that because of the fluid flow pressure drop in the evaporator the temperature at which the juice begins to boil (the temperature to which the juice should be preheated) may be 220°F. or even higher.

Evaporator Design

Calculations are based on the conclusion that at 48.5 GPH and 12.5% vaporization the evaporator of the 48.5 GPH pilot plant used without a preheater as well as the fractionating column are being operated close to maximum as well as optimum conditions; the design is also based on the conclusion that when the evaporator is used with a preheater the length of the evaporator tube should be reduced to one-third its length for best results (cf p. 14). The calculations are also based on the assumption that the 10 GPH unit will be operated at the same juice and steam temperatures as the 48.5 GPH unit; hence, the mean temperature difference for the two units will be the same. The heating load for the 10 GPH unit is 10/48.5 that of the 48.5 GPH unit. The overall heat transfer coefficient is also different, it being assumed that U varies as $V^{0.57}$.

The use of the relationship U varies as $V^{0.57}$ for the evaporator design calculations may be questioned, since this relationship was obtained for the preheater--see Fig. 3. However, since both liquid film and vapor film heat transfer coefficients are correlated by the same basic theoretical equations, and since the preheater U is based on a small diameter tube to which these calculations are directed it is thought that this relationship is the best approximation available. Other factors such as

the effect on the overall heat transfer coefficient of changes in the tube wall thickness or of the tube diameter have been considered but are estimated as having a relatively small effect and have been neglected. When the factors considered above are reduced to their simplest terms it is found that $L = 8.98\ D^{0.14}$ where L is the length in feet of the evaporator tube and D is the I.D. in inches. Fig. 7 is based on this equation.

For a double-tube (tubes in parallel) evaporator the relationships involved are such that the length of each tube, L, given by the above equation must be multiplied by 0.74 and is presented in Fig. 8. Also shown in Figs. 7 and 8 is the estimated time taken for juice passage through the evaporator tube. The time calculation is based on the experimental data that at 48.5 GPH and 10% vaporization this time is 15 seconds, and upon the assumption that the time required is proportional to the volume of the evaporator tube and inversely proportional to the percent vaporized.

A study of Figs. 7 and 8 shows that a double-tube evaporator has no advantage over a single-tube evaporator for small scale processing. (For large scale processing a multiple tube evaporator has an advantage over a single tube because the long length required for a single tube involves mechanical construction difficulties. This length is less for a multiple tube evaporator of the same tube diameter and can be further reduced by the use of smaller diameter tubes.) In fact, since for the same diameter tube the juice passage time through a double-tube evaporator is greater than through a single-tube evaporator, it is recommended that the 10 GPH unit evaporator be a single tube.

Preheater Design

Based on the considerations just given the evaporator design it is found that the preheater tube dimensions are given by the equation:

$$L = 11.9\ D^{0.14}$$

This relationship is shown in Fig. 9.

Fractionating Column Design

The fractionating column height should be kept approximately the same as that for the 48.5 GPH unit; its cross-sectional area can be scaled down directly. For a 10 GPH pilot plant a 1.8 inch I.D. column is thus obtained. The slightly larger size of 2 inch I.D. is recommended, however, in order to take account of the slightly lower free cross-sectional area of smaller sized Raschig rings. Glass beads and other solid types of solid packing should not be used for flooding of the column will probably result.

FIG. 7

EVAPORATOR DIMENSIONS
10 G.P.H., VAPORIZE 12.5% WITHOUT USE OF PREHEATER, ATMOSPHERIC PRESSURE OPERATION, SINGLE HORIZONTAL TUBE.

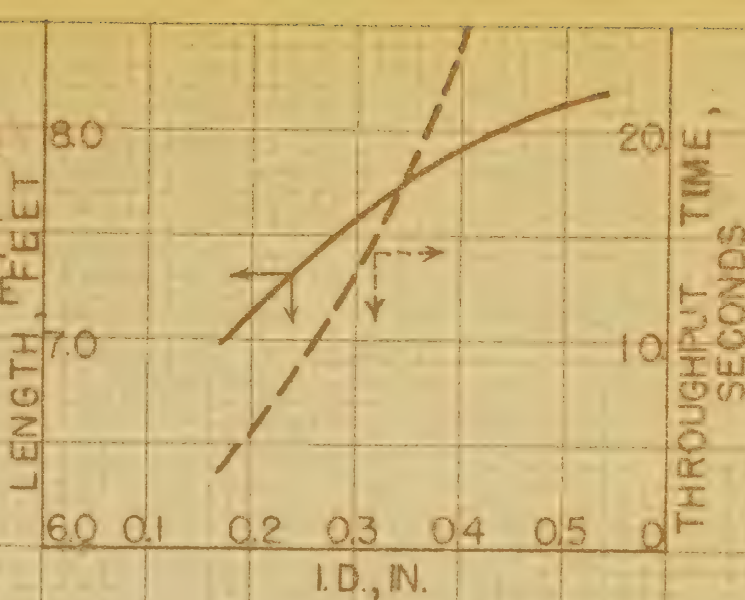


FIG. 8

EVAPORATOR DIMENSIONS
10 G.P.H., VAPORIZE 12.5% WITHOUT USE OF PREHEATER, ATMOSPHERIC PRESSURE OPERATION, DOUBLE HORIZONTAL TUBES ARRANGED IN PARALLEL.

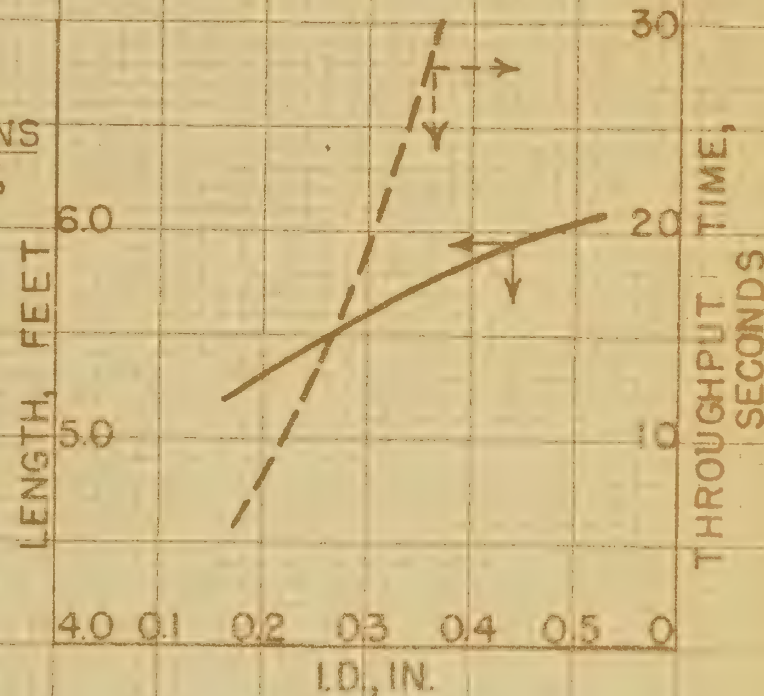
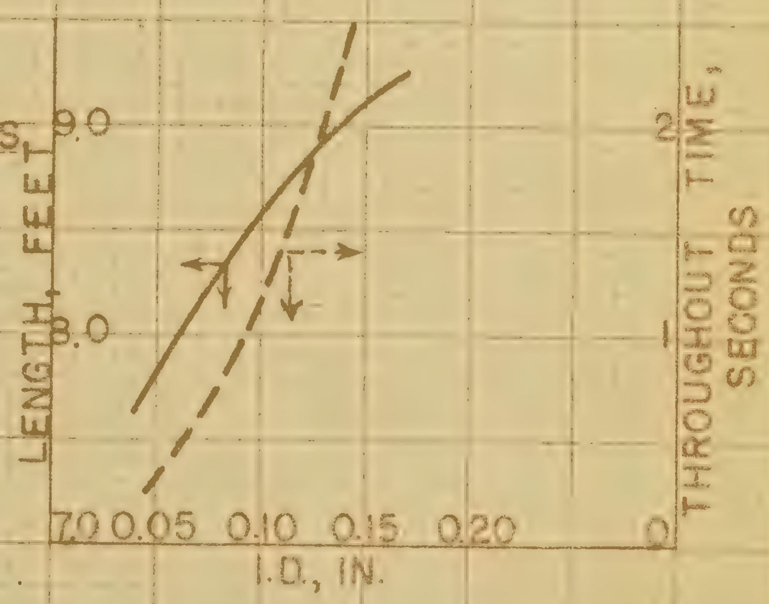


FIG. 9

PREHEATER DIMENSIONS
10 G.P.H., ROOM TEMP. TO 220° F. SINGLE TUBE.



Appendix II

10 GPH PILOT PLANT FOR OPERATING UNDER 27" HIGH VACUUM

Design Suggestions

Resumé

Evaporator--Same size as present 48.5 GPH unit evaporator, horizontal; no preheater; vacuum steam heating may be required.
Superheater -- Use instead of evaporator, superheating the juice up to 260° F and flash-vaporizing it into a chamber maintained at 27 inches of mercury vacuum; for superheater dimensions multiply length given in Fig. 9 by 1.3.
Fractional Distillation Column -- 3 to 4 in. I.D. by a 3-1/2 ft. height of 3/8 inch Raschig ring packing.
Venting System -- Use a wet-type, essence-sealed, vacuum pump and discharge vent gases at atmospheric pressure through a small absorption tower countercurrent to cooled essence.
Permissible leakage -- Not more than 1 inch of mercury per hour for the whole pilot-plant system under operating conditions.

Operating Conditions

The design given is based on processing 10 GPH of juice and vaporizing 12.5% by weight under a vacuum of 27 inches of mercury as measured in the vapor-liquid separator. It is estimated that for the evaporator recommended a heating steam temperature of 180 to 200°F. will be required, hence, a wet-type, condensate vacuum pump should be installed on the condensate drain of the evaporator steam chest. A wet-type vacuum pump is also required on the venting system. Since essence is used as the sealing fluid it is important that the liquid holdup volume of this vacuum pump be kept as small as possible to shorten the time it takes the pilot plant unit to reach steady state conditions. It is suggested that the initial studies be made producing a fifty-fold essence, thus, in effect cutting the holdup volume in half. The more vacuum-tight the pilot-plant unit is constructed the better. In no case should the leakage be greater than 1 inch mercury per hour when the unit is shut off from the vacuum pump (liquid seals at packing glands may be required). Volatile flavor losses in the vent gases and the importance of having a leak-proof unit are discussed in Appendix III.

Evaporator Design

The evaporator design is no longer quite so critical, since the operating temperatures have been lowered. The temperatures, however, have not been lowered as much as superficial observation would indicate. Consider the case of using the evaporator tube on the present 48.5 GPH unit for processing 10 GPH at 27 inches of mercury vacuum. When operated to vaporize 12.5% it is calculated that the pressure drop is 1.5 p.s.i. hence,

the vacuum on the juice being evaporated is 24 inches of mercury, and therefore, the maximum temperature of the juice in the evaporator is approximately 20°F. above the boiling point at 27 inches of mercury vacuum. In order to avoid further increasing this temperature because of a hydrostatic head (1 foot head of juice increases boiling point 6°F. at 27 inches of mercury vacuum) a horizontal tube evaporator is recommended.

Consider further the suitability of the 48.5 GPH pilot plant evaporator for processing 10 GPH under 27 inches of mercury vacuum. The heat load is reduced by 10/48.5, however, it is estimated that the overall heat transfer coefficient is reduced approximately 1/5th (the juice film coefficient decreases by 1/6 to 1/7th, for the product of velocity and density decrease 10/48.5 and the average viscosity of the juice is probably double what it was at atmospheric pressure evaporation).

From these figures it is seen that an evaporator tube having the same dimensions as the one on the present 48.5 GPH unit would probably function satisfactorily. Since approximately the same means temperature difference will be required for the 10 GPH unit as the 48.5 GPH unit, the heating steam temperature required for the 10 GPH unit is estimated to be from 180 to 200°F. Hence, vacuum steam heating will be necessary. Of course, by cutting the length of the evaporator tube in half the estimated required heating steam temperature can be increased to between 220 to 240°F., thus eliminating the necessity for vacuum steam. Such a design is not recommended because the higher tube wall temperatures tend to defeat the purpose of vacuum processing--lower operating temperature.

Superheater Design

The evaporator method of obtaining 10 to 15 percent vaporization is probably superior to the superheater method from a flavor modification standpoint even for vacuum processing, the vapor-liquid separator being maintained under 27 inches of mercury vacuum. A superheater is much easier to construct, however, and a suggested design is therefore given. The preheater designed for use with the evaporator operating at atmospheric pressure was specified to heat 10 GPH of juice from room temperature to approximately 220°F. In order to heat the juice to a temperature of 250° to 260°F. (the temperature at which 12.5% vaporization will result when the juice is flashed into 27 inches of mercury vacuum) the heating steam temperature must be increased. This increase in steam temperature can be minimized by increasing the tube length. It is therefore recommended that the length of the preheater tube given in Fig. 9 be multiplied by 1.3 to obtain the superheater tube length. The heating steam pressure required under these conditions should be less than 40 p.s.i. gauge.

Fractionating Column Design

The capacity of the fractionating column varies as the square root of the (absolute) operating pressure. Hence for 27 inches of mercury vacuum a 4-inch I.D. column would be suitable for a pilot plant pro-

cessing 15 GPH of juice and a 3-inch I.D. column for a pilot plant processing 8.7 GPH. The height and packing of column should be the same as used for the 48.5 GPH atmospheric pressure pilot plant.

Venting System

The necessity for having a venting system whereby the non-condensible gases discharged by the vacuum pump are scrubbed by cool essence at atmospheric pressure is demonstrated in the next section, Appendix III. It is recommended that a small absorption tower be used just as it was for the 48.5 GPH unit--see Fig. 1. Since the discharge from the vacuum pump now contains part of the product, a wet-type, essence-sealed, vacuum pump is necessary.

Two types of wet vacuum pumps are commercially available: (1) rotary, and (2) reciprocating. The first type is represented by the Nash Hytor pump, manufactured by the Nash Engineering Co., South Norwalk, Connecticut. The capacity of this type of pump falls off rapidly above 25 inches of mercury vacuum so that it is customary to use a two-stage arrangement for 27 inches of mercury vacuum. However, the capacity of the smallest pump that Nash makes is so much in excess of what is required for this necessarily nearly vacuum-tight system that a single stage would probably be satisfactory. (Nash's new small size pump, Model 332, which has an overall diameter of about 4 inches, is available only on special order at present.) A small wet-type, reciprocating pump (4 strokes per second) is made by the Ernest Scott and Co., Fall River, Massachusetts. Although this model, size 3 x 3, is a rather cumbersome pump compared to the Nash Hytor, its design probably could be modified so that its liquid hold-up volume would be smaller. In any event both of these pumps merit careful consideration before one is ordered to operate a volatile flavor-recovery plant under vacuum.

Appendix III

VENT LOSS CALCULATIONS FOR A VACUUM PROCESS

In transforming an ordinary evaporation and/or distillation process, such as the rectification of alcohol-water mixtures, from atmospheric pressure operation to vacuum, the vent losses are increased but in any case are negligible as long as the condensate temperature is maintained at a safe margin below the vapor condensation temperature. The same is not true, however, for the volatile flavor recovery process, because the volatile flavors in the essence condensate even though concentrated 100 or 200 times with respect to the fresh juice, are present only to an estimated few tenths of one percent by weight and have a high vapor pressure even at the freezing point of the condensate. An appreciation of this fact as well as of the stoichiometric relationships involved as outlined below will lead to an understanding of why previous investigators failed to obtain essences much above 10 to 20 fold without incurring appreciable losses of volatile flavor.

Nomenclature

V mols of non-condensible gases evolved per hour from juice
F mols of volatile flavor in the vent gas
P operating pressure in inches of mercury
p partial pressure of volatile flavors in 100-fold essence, inches of mercury
L mols of volatile flavor lost in the vent gas

Subscripts

27 vacuum operation (27 inches of mercury in the examples)
A atmospheric pressure operation

Comparison of Vent Loss at 27 inches of Mercury and Atmospheric Pressure Operation

L = (total mols of vent gas) (mol fraction of volatile flavors)

Assuming no leakage of air into the process,

$$L = \left[\frac{V}{\frac{P - p}{P}} \right] \frac{p}{P} = \frac{Vp}{P-p}$$

Assume that p = 1 inch of mercury at the vent temperature which is held somewhere in the range of 32°F. to 70°F. (Note vapor pressure of water = 1 inch of mercury at 79°F). The loss of volatile flavors in the vent gas at atmospheric pressure and vacuum operation is as follows:

$$L_A = \frac{V x 1}{30-1} = 0.0345V$$

$$L_{27} = \frac{V x 1}{3-1} = 0.5000V$$

Difference 0.4655V

Thus, the loss of volatile flavors for vacuum processing is increased 14.4 times. In order to recover this loss it is proposed that a wet-type, essence-sealed, vacuum pump be used and that the vent gases discharged from this vacuum pump be scrubbed at atmospheric pressure with essence in an absorption tower.

Leakage Tolerable under Vacuum Operation

It is obvious that since the leakage in effect increases V, the volatile flavor loss will be increased proportionately even though the vent system just described is used. For orange juice the rate of noncondensible gas evolved from the juice was found to be 5% by volume (this figure may be different for orange juice obtained the first part of the season). At 10 GPH of juice this amounts to 0.068 cu. ft. (S.T.P.). Suppose that loss

due to leakage is compensated for by producing a 50-fold essence instead of a 100-fold, the amount of leakage that can be tolerated will now be solved for. Assume that Henry's law applies, hence p = 0.5 inches of mercury. Let W represent the mols of air leakage into the system which is permissible. The total mols of noncondensible gas is now (V + W). The loss of volatile flavor from the vent system is therefore given by the equation

$$L = 0.0345V = \frac{(V + W)\, 0.5}{30 - 0.5}$$

whence W = 1.01 V, and since V in cubic feet (S.T.P.) is 0.068 the leakage that can be tolerated is 0.069 cu. ft. (S.T.P.). The volume of a 10 GPH unit for vacuum operation is estimated as 2 cu. ft. The drop in vacuum which can be tolerated, measured when the vacuum pump is shut off, is therefore $30 \times \frac{0.069}{2} = 1.0$ inch of mercury per hour.

New Scheme for Processing Under Vacuum

In order to avoid the somewhat troublesome use of a wet-type, essence-sealed vacuum pump, a method of processing has been devised as follows (all steps under vacuum):

(1) Vaporize 12.5% under vacuum, separate vapors from liquid, and condense the vapors, discarding the vent gases through an "ordinary" type of vacuum pump;

(2) Rectify the condensate obtained from step (1) in a fractionating column operated under vacuum, and again discard the now negligible amount of vent gases through an "ordinary" type of vacuum pump.

Vent loss calculations similar to those just given will demonstrate that for such a process to be successful practically all and not just 87.5% of the non-condensible gases evolved by the juice must be eliminated in step (1) and that the fractionating column system must be nearly leak-proof--less than 0.2 inch of mercury per hour. Although this scheme does not look very promising on paper it has been described because it may be worthwhile testing experimentally.

Appendix IV

COEFFICIENT OF HEAT TRANSFER FOR THE EVAPORATOR OPERATED WITHOUT A PREHEATER

The thermal and related data on the evaporator performance which was taken for each runwere as follows:

GPH of Juice,
Vaporization - Percent by weight,
Juice temperature in and out,
Heating steam temperature.

In the first or preheater section of the evaporator tube the juice is heated from room temperature up to its boiling point. The temperature of the juice then remains relatively constant, decreasing only a few degrees farenheit corresponding to the pressure drop of the juice flow through the vaporization section of the evaporator. The basic heat transfer equation, $Q_E = U_E A_E T_E$ applied to the whole evaporator tube is therefore meaningless because of the impossibility of properly evaluating T_E from the available data. However, by redefining U_E it becomes possible to compute values of U_E as well as U_pA_p and U_VA_V and in turn to study the effect of operating variables on them.

Nomenclature

Q	heat transferred, BTU/hour	
U	overall heat transfer coefficient	$\frac{(BTU)}{(Hr)(Sq. Ft.)(^\circ F.)}$
A	area, sq. ft.	
T	mean temperature difference, °F.	

Subscripts

E	entire evaporator tube
P	preheater section of evaporator tube from inlet to point at which juice reaches its maximum temperature
V	vaporization section of evaporator tube, that is, $A_V = A_E - A_P$

U_E is defined by the equation $U_EA_E = U_PA_p + U_VA_V$.

Since $U_p = \frac{Q_p}{A_pT_p}$ and $U_V = \frac{Q_V}{A_VT_V}$

then $U_pA_p = \frac{Q_p}{T_p}$ and $U_VA_V = \frac{Q_V}{T_V}$

Also it follows that

$$U_E = \frac{Q_p}{A_ET_p} + \frac{Q_V}{A_ET_V}$$

Note that all the data necessary to compute U_E, U_PA_P and U_VA_V is available. It is also possible to compute values of A_p and A_E by noting the change of U_pA_p and U_VA_V caused by varying the operating conditions, estimating the effect of the change of the operating conditions upon U_P and U_V and then solving for A_p and A_V with the aid of the simultaneous equations $U_EA_E = U_PA_P + U_VA_V$ and $A_E = A_p + A_V$, in which U_E and A_E are known. Although it is felt that such an analysis is beyond the scope of this report it is mentioned here for possible use in future studies. Values of U_E, U_pA_p, U_VA_V, T_p and T_V from runs of experiment 44R4 are tabulated below. T_p is the log mean temperature difference. (The maximum juice temperature in the evaporator tube at 48.5 GPH and 10% vaporization was estimated from the pressure drop to be 223°F. At lower flow rates and therefore lower pressure drops this maximum temperature is correspondingly lower.)

GPH	% Vaporization	U_E	U_pA_p	U_VA_V	T_p	T_V
Water						
48.5	10.5	621	647	1018	87	38
Orange Juice						
48.5	5.3	455	584	636	78	33
48.5	7.9	533	572	858	82	36
48.5	10.1	573	558	978	86	40
24.3	9.7	484	363	935	64	21
12.2	10.0	521*	255*	1142*	46*	9*

Examination of the experimental data discloses that the values marked with an may be in error as much as 20%

The T's are tabulated because in studying the effect of operating variables on U_pA_p, U_VA_V, and U_E it is important to note that the percentage change in T_p is different from that of T_V.

Appendix V

TABULATION OF HEAT TRANSFER DATA

Evaporator Data

Experiment No.	Consecutive Minutes on Juice	G.P.H.	Wt. Per Cent Vaporized	Temperature, °F. Juice in	Estimated Maximum	Juice Out	Steam
44R1	(On Water)	48.5	10.0	84	223	217	258
"	8	48.5	10.0	84	224	217	262
44R2	(On Water)	48.5	8.0	85	219	215	243
"	17	48.5	7.7	86.5	221	217	250
"	40	48.5	7.7	87	221	217	253.5
44R3	(On Water)	48.5	9.1	85	221	216	250
"	10	48.5	8.7	90	222	217	257
"	115	48.5	8.8	90	222	217	255.5
"	130	48.5	5.2	90	218	216	246
"	130+	48.5	10.1	90	223	217	262
"	130+	36.7	9.7	90	220	216	246
"	130+	24.3	10.0	90	217	215	231
44R4	(On Water)	48.5	10.5	84	223	217	258
"	5	48.5	10.1	92.5	223	217	260
"	10	48.5	7.9	93	220	216	254
"	20	48.5	5.3	92.8	217	215	249
"	40	24.3	9.7	91.5	217	216	237.5
"	60	12.2	10.2	91	217	216	225.7
"*	80	48.5	10.0	217*	223	217.8	239.5
44R5	(On Water)	39.7	13.0	83	223	216	251.5
"	10	39.7	12.3	66	223	217	259
"	105	39.7	12.3	67	223	217.5	259.7
"	110	48.5	10.0	67	223	217.5	263
44R6	(On Water)	40	12.9	83	223	217.5	252.5
"	10	40	12.5	90	223	218	256
"	150	40	12.5	91	223	218	256
44R7	(On Water)	40	12.9	84	223	218	251.5
"	5	40	12.2	89	223	218	258

(*Preheater used)

Preheater Data

Expt. No.	Consecutive Minutes on Juice	GPH	Temperatures °F. Juice in	Juice Out	Steam, Thermo-couple	Steam, Press. Gauge	Steam, in Calc.
44R4	10	48.5	91	217*	239.5	240.5	240
"	50	39.5	91	282	295	297.6	297
"	60	39.5	91	210.5	222	232	228
"	70	48.5	91	211	229.5	239	235
"	80	12.2	91	211.7	214	215.3	215
"	90	6.1	91	212.1	215.8	214.6	214.5

* Evaporator used on this run only.

Cooler Data--Stainless Steel Water Jacketed Unit

Expt. No.	Consecutive Minutes on Juice	Juice Lbs/Hour	Temperatures Juice In	Juice Out	Cooling Water In
44R1	(Water)	364	217	105	84
"	8	380	218	118	84
44R2	(Water)	372	215	106	85
"	17	389	216.5	119	85
44R3	10	385	217	113	85
"	75	385	217	116	85
"	130	400	216	116	85
"	140	380	217	110	85

Appendix VI

COOLER OVERALL HEAT TRANSFER CALCULATIONS

Neither the cooling water rate nor the cooling water exit temperature were measured in these experiments, and one of these values must be known in order to compute the overall heat transfer coefficient. It is estimated, however, that the cooling water rate was between 2 to 6 gallons per minute. If now the exit cooling water temperature is calculated by means of a heat balance on the cooler the log mean temperature difference for the range of conditions represented by 2 to 6 gallons per minute of cooling water can be represented as T $\pm$ 10% which is well within the experimental error. The average value of the overall heat transfer coefficients computed for the data given in Appendix V is 253 BTU/(Hr.)(Sq. ft.)(°F); the average deviation from 253 is $\pm$ 26, the mean is $\pm$ 9.

Appendix VII

FLAVOR EVALUATION TESTS ON UTILIZATION OF ORANGE ESSENCE

Preparation of Concentrates June 5, 1945 with and without Essence for Storage Tests

26.75 lbs. orange concentrate; 62.6% solids added; 0.02675 lbs. of sodium benzoate dissolved in 40 ml. of water; mixed by stirring. The mixed concentrate was divided into two parts and treated as follows:

#1. 13.5 lbs. concentrated, added 350 ml. of 200X orange essence (44R3 essence) and mixed, final brix 61°.

#2. 13 lbs. concentrate, added 340 ml. of distilled water and mixed, final brix 60°.

The concentrate was packed in enamelled and plain tin cans, size 211 x 210.
12 cans enamelled with essence marked EE.
12 cans plain tin with essence marked PE.
12 cans plain tin control marked PC
12 cans enamelled control marked EC

Organoleptic Tests September 14, 1945

One can of each lot of orange concentrate canned on June 5, was opened and diluted to 12 Brix for taste and aroma tests. Tasters were Veldhuis, Patrick, Curl, Swift and Atkins. The color of the concentrate was graded from a natural color for that from 37°F. storage to a brown for the sample from 90°F. storage. The samples which were kept at 80° and 90° were discolored and gasing badly, were judged to be unpalatable and so were discarded. The samples which had been stored at 37° and 60°F. were tested for taste and aroma with the following results. Each taste tester was asked to taste the samples and then to arrange them in the order of their preference. The samples were coded so that the identity of the samples was not known.

Evaluation of 37°F Storage Sample

	Judges' Order of Preference				
	A	B	C	D	E
1st best	No. 4	No. 2	No. 3	No. 4	No. 4
2nd best	3	3	4	3	3
3rd best	1	4	1	1	2
4th poorest	2	1	2	2	1

Evaluation of 60°F. Storage Samples

	A	B	C	D	E
	Judges' Order of Preference				
1st best	No. 4	No. 2	No. 1	No. 4	No. 1
2nd best	3	3	2	3	4
3rd best	1	1	3	1	3
4th poorest	2	4	4	2	2

Sample No.

1 canned in plain tin no essence added
2 canned in enamelled tin, no essence added
3 canned in enamelled tin, essence added
4 canned in plain tin, essence added

Peel Oil and Essence Emulsion Tests

Emulsion 1	orange peel oil	0.4 ml.
	200X orange essence	10.0 ml.
Emulsion 2	Emulsone B	0.06 gms.
	diluted to 25 ml.	

1.25 ml. of emulsion (1) was added to 100 ml. of distilled water. 1.25 ml. of emulsion (2) was added to another 100 ml of distilled water. After mixing the judges were requested to determine which had an aroma most characteristic of orange juice. The judges voted 3-2 for the sample containing peel oil and essence.

A sample of orange concentrate was diluted with distilled water, final brix 12.0. 200 ml. of the diluted concentrate was placed into each of four beakers. Beaker No. 1 was used as a control; orange peel oil emulsion was added to beaker No. 2; orange peel oil and essence emulsion was added to No. 3; and essence only was added to Beaker No. 4. Five judges, after smelling and tasting the four samples, voted as follows:

	A	B	C	D	E
1st best	No. 4	No. 3	No. 4	No. 4	No. 3
2nd best	2	2	2	1	2
3rd best	3	4	3	3	4
4th poorest	1	1	1	2	1

Score No. 1, 7 points; No. 2, 13; No. 3, 14; No. 4, 16

No. 1 - control
2 - orange peel oil emulsion added
3 - peel oil and essence emulsion added
4 - orange essence added

Orange essence, 200X and 400X, after four months room temperature storage had undergone a definite deterioration when compared to essence stored at 37° F.

Appendix VIII

GRAPEFRUIT JUICE ESSENCE RECOVERY

Objective:

Total volatile flavor recovery from grapefruit juice. Preparing 100X essence using oil-trap decanter on condenser line so that both reflux and essence are single phase.

Grapefruit Juice Used:

Brix	12.6%
Acid	1.56%
Peel oil raw juice	0.008%
Peel oil stripped juice	0.004%

Procedure:

Same as for orange juice runs: feed rate was 39.7 GPH and vaporization was 10%.

Remarks:

One five-gallon (18900 ml.) bottle of stripped juice was collected in seven minutes ≠ five seconds. Oil separator not working as well as with orange. Very little oil collected in trap. Some oil came over with essence. Peel oil in original juice 0.008% by vol., peel oil in stripped juice 0.004%.

Taste and Aroma Test: Veldhuis, Patrick, Curl, Swift, Pulley

Aroma:	Vote
Fresh juice has best aroma	5
Stripped juice has best aroma	0
Juice with added essence best	0

Taste:

Fresh juice best	2
Stripped juice best	2
Juice with essence best	1

Two of the tasters thought that the addition of essence gave the juice a fermented taste and aroma.

Data:

Feed rate 39.7 G.P.H.
Small pump setting 0.00, large pump 3.10
Vaporization, 10%

Variac Settings:

Separator, 105V; vapor line 80V; reboiler 50V; setting for bellows pump 26.1 ml/min.; run on total reflux for 7½ min.; manometer 18-19 in. water.

Juice started 2:30 p.m., juice at separator 2:31 p.m.
Bellows pump started 2:39 p.m.
Bellows pump calibration, 25.2, 25.5 ml/min.
Vent gas measurement, 30 cc./6 sec.
Juice stopped 3:18 p.m.

Temperatures	Water	Water	Juice 2:39 P.M.	Juice 2:58 P.M.	Juice 3:15 P.M.
Juice to Evaporator	86	86	83	83.5	85
Evaporator Steam Chest	243	243	250	250	251
Separator	216	216	215	215	215
Vapor to Fractionating Column	214.5	214.5	214	214	214
Condensate	115.0	115	114	121	123
Stripped Juice			129°F		
Manometer inches of water	18	18	18	18	18
Steam pressure p.s.i.	12.5	12.5	16¼	16¼	17
Juice pressure p.s.i.	21½	22	26	26	26

Product and Samples Collected

Essence 2 oz. bottles

45R1-1	2:45
2	2:48
3	2:51
4	2:54
5	2:58
6	3:00
7	3:05
8	3:09
9	3:11
10	
11	3:16

12 & 13 pumped out of disconnected trap

Stripped juice 45R1-51A, 2:55, B.C,D

Bot. prod. 45R1-91, A,B,C,D

Fresh juice 45R1, A,B,C,D

Appendix IX

SUPPLEMENT TO CIRCULAR AIC-63

Since Circular AIC-63 entitled "Recovery and Utilization of Natural Apple Flavors" was published, in September 1944, additional information has been obtained pertinent to the design of equipment for the process.

To insure complete recovery of the aroma, the first step of the process necessitates the vaporization of 10 percent of the fresh juice. The heating and vaporization must be done rapidly enough to avoid modification of the fresh flavor. In the process described in the circular this was accomplished by superheating the juice to 320° F. in 3 seconds and then flashing to atmospheric pressure.

On pages 4, 8 and 10 of the circular it is suggested that a possible alternate method would be to heat and vaporize the juice by a single passage through a rapid evaporator. Recent experiments have indicated that for industrial application of the process a rapid evaporator is superior to a superheater. The most important advantages of the evaporator method are that the fouling of the heating surfaces by the juice is reduced to a small fraction of what occurs in the superheater and that steam pressures of only about 30 lbs. per sq. in. (gauge) are needed, whereas up to 120 lbs. per sq. in. (gauge) are required for the superheater. The evaporator method, as well as the superheater method, sterilizes the juice as well as the essence.

This information is based on pilot-plant experience with a mixture of McIntosh and Stayman Winesap apples, fully ripe. Unfiltered juice was used because it represents the most unfavorable conditions that would be encountered in practice with respect to fouling of the tubes. The inside diameter of the evaporator tube was 0.62 inch; its heated length was 16-1/2 feet; and 30 lbs. per sq. in. of steam was used in the steam chest. Under these conditions the juice could be heated for a total time of at least 15 seconds without modification of the fresh flavor, and the rate of processing the juice was 50 gallons per hour. The modification of flavor resulting from doubling this heating time would not be very significant. However, in general it is desirable to heat the juice for as short a time as possible, consistent with practical evaporator design.

Under certain conditions, as for example when the maximum steam pressure available is much less than 30 lbs. per sq. in., it may be desirable to employ a high velocity preheater. However, when such a preheater with a tube of 0.18 inch inside diameter was used ahead of the evaporator described above, the total permissible heating time without modification of fresh flavor was only 10 seconds when the time for preheating the juice to its boiling point was 2.0 seconds.

With the evaporator system, either alone or with a preheater, fouling is so slight that daily cleaning would probably be sufficient. Moreover, we have been able to remove in a few minutes the fouling deposit formed in a 3-hour run on the tubes of both the preheater and the evaporator. This is accomplished by shutting off the steam, pumping cold water through the heating tubes, then stopping the pumps and suddenly admitting steam to the steam chest. The boiling, that takes place inside the tubes shortly thereafter, removes the deposit.